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4-(Octyloxy)phenyl 2-oxo-2H-chromene-3-carboxylate

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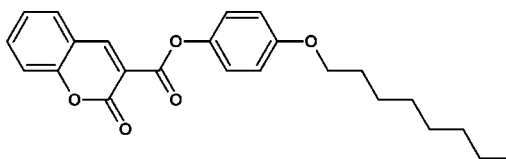
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.058; wR factor = 0.146; data-to-parameter ratio = 13.7.

In the title compound, $\text{C}_{24}\text{H}_{26}\text{O}_5$, the 2H-chromene ring system is essentially planar, with a maximum deviation of 0.029 (2) Å from the best-fit mean plane incorporating both rings. The dihedral angle between the 2H-chromene ring system and the benzene ring is 21.00 (1)°. In the crystal, pairs of $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds generate an $R_2^2(8)$ ring pattern. These contacts are bolstered by weaker bifurcated $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background to coumarin derivatives and their biological and technological applications, see: Georgieva *et al.* (2004); Creaven *et al.* (2005); Morita *et al.* (2005); Tian *et al.* (2003); Iliopoulos *et al.* (2010); Hejchman *et al.* (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_{24}\text{H}_{26}\text{O}_5$
 $M_r = 394.45$
 Monoclinic, $P2_1/n$
 $a = 14.464$ (3) Å
 $b = 6.7548$ (15) Å
 $c = 21.381$ (5) Å
 $\beta = 91.663$ (8)°

 $V = 2088.0$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.29 \times 0.25 \times 0.21$ mm

Data collection

 Bruker SMART CCD area-detector
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2007)
 $T_{\min} = 0.975$, $T_{\max} = 0.982$

 22609 measured reflections
 3615 independent reflections
 1926 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.084$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.146$
 $S = 0.94$
 3615 reflections

 264 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C4}-\text{H4}\cdots\text{O1}^i$ | 0.93 | 2.59 | 3.513 (4) | 174 |
| $\text{C9}-\text{H9}\cdots\text{O2}^i$ | 0.93 | 2.51 | 3.420 (3) | 167 |
| $\text{C16}-\text{H16}\cdots\text{O2}^i$ | 0.93 | 2.71 | 3.551 (3) | 151 |
| $\text{C16}-\text{H16}\cdots\text{O3}^i$ | 0.93 | 2.63 | 3.338 (3) | 133 |

Symmetry code: (i) $x, y + 1, z$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 2012); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5291).

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supporting information

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4-(Octyloxy)phenyl 2-oxo-2*H*-chromene-3-carboxylate

B. S. Palakshamurthy, S. Sreenivasa, H. T. Srinivasa, K. R. Roopashree and H. C. Devarajegowda

S1. Comment

Coumarin derivatives have been attracted increasing attention due to their extensive biological applications, such as anticancer, anti-inflammatory and anticoagulant agents (Georgieva *et al.*, 2004; Creaven *et al.*, 2005). The coumarin nucleus has been the focus of our recent research concerning the design, synthesis and characterization to investigate their liquid crystal properties together with crystal structure studies (Morita *et al.*, 2005; Tian *et al.*, 2003). Coumarins are interesting class of heterocycles because of their dipolar moment increases by external stimulus such as light, temperature, electric current and chemical reaction (Iliopoulos *et al.*, 2010). The excitation of the coumarin chromophore increases the electron density of its carbonyl groups owing to excited photochemical and photophysical properties such as molecular fluorescent sensors, laser dyes and many industrial applications (Hejchman *et al.*, 2011).

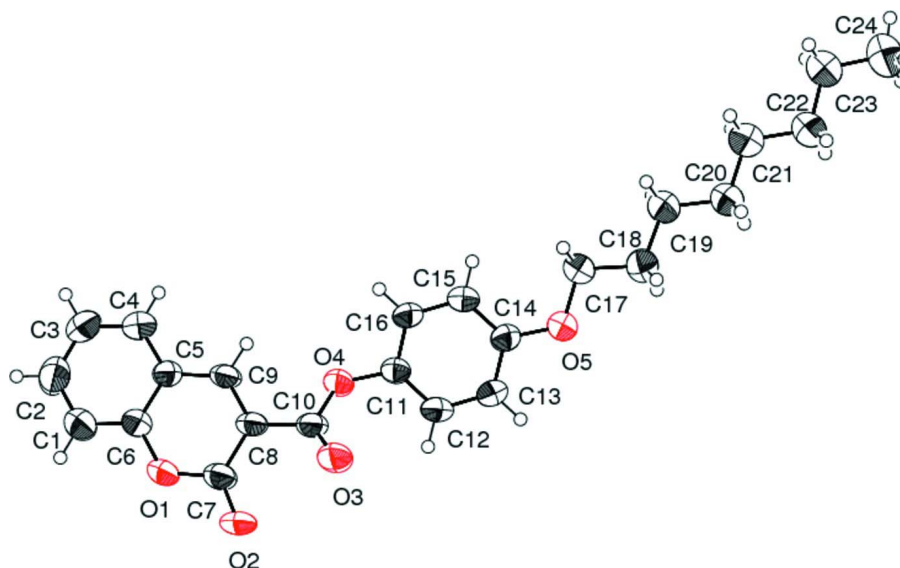
The asymmetric unit of 4-(octyloxy)phenyl 2-oxo-2*H*-chromene-3-carboxylate is shown in Fig. 1. The 2*H*-chromene ring (O1/C1–C9) system is planar, with a maximum deviation of 0.028 (2) Å for atom C8. The dihedral angle between 2*H*-chromene ring (O1/C1–C9) and benzene ring (C11–C16) is 21.11 (1)°. The crystal structure is characterized by intermolecular C4—H4···O1 and C9—H9···O2 hydrogen bonding generating an $R_2^2(8)$ ring pattern (Bernstein *et al.*, 1995). Bifurcated C16—H16···O2 and C16—H16···O3 contacts further strengthen the packing, Fig. 2.

S2. Experimental

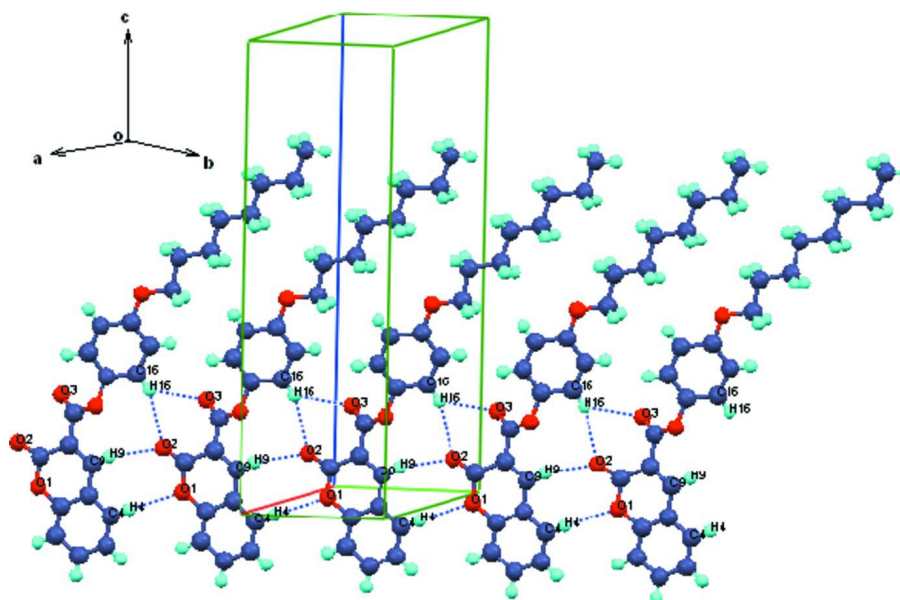
A mixture of 2-oxo-2*H*-chromene-3-carboxylic acid (19 mg, 1 mmol), 4-(octyloxy)phenol (22.2 mg, 1 mmol), *N,N*-dicyclohexylcarbodiimide (23 mg, 1.2 mmol) and a catalytic quantity of *N,N*-dimethylaminopyrimidine was stirred in 5 ml of dry dichloromethane for 24 h at room temperature. The residue obtained on removal of solvent was chromatographed on silica gel and eluted with chloroform. Removal of solvent from the eluate afforded a colorless solid, which was recrystallized from absolute ethanol to obtain needle like crystals of the title compound for X-ray diffraction analysis.

S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H, C—H = 0.97 Å for methylene H and C—H = 0.96 Å for methyl H, and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for all other H.

**Figure 1**

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.

**Figure 2**

Crystal packing for the title compound with hydrogen bonds drawn as dashed lines.

4-(Octyloxy)phenyl 2-oxo-2*H*-chromene-3-carboxylate

Crystal data

$C_{24}H_{26}O_5$

$M_r = 394.45$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 14.464\ (3)\ \text{\AA}$

$b = 6.7548\ (15)\ \text{\AA}$

$c = 21.381\ (5)\ \text{\AA}$

$\beta = 91.663\ (8)^\circ$

$V = 2088.0\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 840$
 $D_x = 1.255 \text{ Mg m}^{-3}$
 Melting point: 580 K
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 3615 reflections

$\theta = 3.2\text{--}25.0^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Needles, colourless
 $0.29 \times 0.25 \times 0.21 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω and ϕ scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2007)
 $T_{\min} = 0.975$, $T_{\max} = 0.982$

22609 measured reflections
 3615 independent reflections
 1926 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.084$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -17 \rightarrow 16$
 $k = -8 \rightarrow 5$
 $l = -25 \rightarrow 25$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.146$
 $S = 0.94$
 3615 reflections
 264 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.14 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0019 (7)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|--------------|-------------|---------------|----------------------------------|
| O1 | 0.11082 (14) | 0.0294 (3) | −0.01675 (8) | 0.0705 (6) |
| O2 | 0.11756 (15) | −0.1104 (3) | 0.07552 (9) | 0.0826 (7) |
| O3 | 0.06844 (14) | 0.1538 (3) | 0.17685 (9) | 0.0744 (6) |
| O4 | 0.14523 (12) | 0.4373 (2) | 0.16091 (7) | 0.0610 (5) |
| O5 | 0.14206 (14) | 0.7392 (3) | 0.40100 (8) | 0.0730 (6) |
| C1 | 0.1075 (2) | 0.1595 (5) | −0.11861 (13) | 0.0755 (9) |
| H1 | 0.1043 | 0.0314 | −0.1345 | 0.091* |
| C2 | 0.1090 (2) | 0.3196 (5) | −0.15818 (14) | 0.0827 (9) |
| H2 | 0.1062 | 0.2995 | −0.2012 | 0.099* |
| C3 | 0.1146 (2) | 0.5101 (5) | −0.13485 (14) | 0.0775 (9) |

| | | | | |
|------|--------------|------------|---------------|-------------|
| H3 | 0.1165 | 0.6174 | −0.1620 | 0.093* |
| C4 | 0.11723 (19) | 0.5398 (4) | −0.07203 (13) | 0.0682 (8) |
| H4 | 0.1203 | 0.6682 | −0.0564 | 0.082* |
| C5 | 0.11538 (17) | 0.3810 (4) | −0.03071 (11) | 0.0509 (6) |
| C6 | 0.11088 (18) | 0.1917 (4) | −0.05564 (12) | 0.0557 (7) |
| C7 | 0.11337 (19) | 0.0428 (4) | 0.04771 (12) | 0.0603 (7) |
| C8 | 0.11338 (17) | 0.2423 (3) | 0.07410 (11) | 0.0497 (6) |
| C9 | 0.11632 (16) | 0.3995 (4) | 0.03580 (11) | 0.0524 (7) |
| H9 | 0.1191 | 0.5255 | 0.0533 | 0.063* |
| C10 | 0.10603 (18) | 0.2654 (4) | 0.14224 (12) | 0.0531 (7) |
| C11 | 0.13845 (18) | 0.5046 (4) | 0.22323 (11) | 0.0535 (7) |
| C12 | 0.16625 (19) | 0.3932 (4) | 0.27383 (12) | 0.0639 (8) |
| H12 | 0.1845 | 0.2623 | 0.2686 | 0.077* |
| C13 | 0.16691 (19) | 0.4769 (4) | 0.33248 (12) | 0.0650 (8) |
| H13 | 0.1863 | 0.4028 | 0.3671 | 0.078* |
| C14 | 0.13875 (19) | 0.6712 (4) | 0.34023 (11) | 0.0567 (7) |
| C15 | 0.11116 (19) | 0.7799 (4) | 0.28884 (11) | 0.0619 (7) |
| H15 | 0.0923 | 0.9104 | 0.2938 | 0.074* |
| C16 | 0.11120 (19) | 0.6972 (4) | 0.22990 (11) | 0.0591 (7) |
| H16 | 0.0929 | 0.7715 | 0.1951 | 0.071* |
| C17 | 0.1323 (2) | 0.9462 (4) | 0.41084 (12) | 0.0645 (7) |
| H17A | 0.0739 | 0.9916 | 0.3926 | 0.077* |
| H17B | 0.1819 | 1.0168 | 0.3909 | 0.077* |
| C18 | 0.1356 (2) | 0.9869 (4) | 0.47999 (11) | 0.0675 (8) |
| H18A | 0.1914 | 0.9287 | 0.4984 | 0.081* |
| H18B | 0.0831 | 0.9239 | 0.4989 | 0.081* |
| C19 | 0.1343 (2) | 1.2047 (4) | 0.49476 (12) | 0.0707 (8) |
| H19A | 0.0812 | 1.2634 | 0.4731 | 0.085* |
| H19B | 0.1893 | 1.2646 | 0.4780 | 0.085* |
| C20 | 0.1304 (2) | 1.2574 (4) | 0.56327 (12) | 0.0723 (8) |
| H20A | 0.0761 | 1.1960 | 0.5806 | 0.087* |
| H20B | 0.1843 | 1.2027 | 0.5850 | 0.087* |
| C21 | 0.1269 (2) | 1.4752 (5) | 0.57543 (13) | 0.0831 (9) |
| H21A | 0.0717 | 1.5278 | 0.5546 | 0.100* |
| H21B | 0.1797 | 1.5360 | 0.5562 | 0.100* |
| C22 | 0.1265 (2) | 1.5383 (5) | 0.64278 (13) | 0.0839 (9) |
| H22A | 0.1818 | 1.4883 | 0.6641 | 0.101* |
| H22B | 0.0734 | 1.4801 | 0.6625 | 0.101* |
| C23 | 0.1229 (3) | 1.7629 (5) | 0.65022 (15) | 0.1031 (12) |
| H23A | 0.1746 | 1.8199 | 0.6287 | 0.124* |
| H23B | 0.0666 | 1.8108 | 0.6295 | 0.124* |
| C24 | 0.1255 (3) | 1.8364 (6) | 0.71544 (16) | 0.1332 (16) |
| H24A | 0.0720 | 1.7894 | 0.7365 | 0.200* |
| H24B | 0.1256 | 1.9785 | 0.7153 | 0.200* |
| H24C | 0.1804 | 1.7889 | 0.7368 | 0.200* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| O1 | 0.1120 (16) | 0.0356 (11) | 0.0641 (12) | −0.0062 (10) | 0.0039 (10) | −0.0070 (9) |
| O2 | 0.1353 (19) | 0.0309 (12) | 0.0816 (14) | −0.0007 (11) | 0.0057 (12) | 0.0064 (10) |
| O3 | 0.1055 (16) | 0.0459 (12) | 0.0723 (12) | −0.0202 (11) | 0.0096 (11) | 0.0020 (10) |
| O4 | 0.0867 (14) | 0.0398 (11) | 0.0564 (11) | −0.0166 (9) | 0.0003 (9) | −0.0004 (9) |
| O5 | 0.1129 (17) | 0.0487 (13) | 0.0570 (12) | 0.0036 (10) | −0.0023 (10) | −0.0017 (9) |
| C1 | 0.103 (2) | 0.059 (2) | 0.0653 (19) | −0.0103 (16) | 0.0005 (16) | −0.0075 (16) |
| C2 | 0.098 (3) | 0.090 (3) | 0.0596 (18) | −0.0053 (19) | −0.0046 (16) | 0.0027 (19) |
| C3 | 0.097 (3) | 0.066 (2) | 0.069 (2) | 0.0052 (17) | 0.0010 (16) | 0.0184 (17) |
| C4 | 0.082 (2) | 0.0507 (18) | 0.0718 (19) | 0.0039 (15) | 0.0006 (15) | 0.0067 (16) |
| C5 | 0.0562 (17) | 0.0401 (16) | 0.0562 (15) | 0.0004 (12) | −0.0026 (12) | 0.0034 (13) |
| C6 | 0.0659 (19) | 0.0445 (17) | 0.0566 (16) | −0.0040 (13) | −0.0021 (13) | −0.0030 (14) |
| C7 | 0.075 (2) | 0.0386 (17) | 0.0672 (18) | −0.0039 (13) | 0.0036 (14) | −0.0036 (15) |
| C8 | 0.0573 (17) | 0.0307 (14) | 0.0608 (16) | −0.0013 (11) | −0.0018 (12) | 0.0018 (13) |
| C9 | 0.0623 (18) | 0.0326 (15) | 0.0622 (16) | 0.0006 (12) | −0.0020 (12) | −0.0042 (12) |
| C10 | 0.0590 (17) | 0.0361 (16) | 0.0639 (17) | −0.0016 (13) | −0.0025 (13) | 0.0025 (14) |
| C11 | 0.0697 (19) | 0.0365 (15) | 0.0542 (15) | −0.0078 (12) | 0.0018 (12) | 0.0008 (13) |
| C12 | 0.087 (2) | 0.0373 (15) | 0.0670 (18) | 0.0048 (14) | −0.0062 (15) | 0.0005 (14) |
| C13 | 0.092 (2) | 0.0435 (17) | 0.0588 (17) | 0.0071 (14) | −0.0091 (14) | 0.0103 (14) |
| C14 | 0.0749 (19) | 0.0434 (17) | 0.0515 (16) | −0.0048 (13) | −0.0026 (13) | 0.0046 (13) |
| C15 | 0.088 (2) | 0.0377 (15) | 0.0599 (17) | 0.0002 (14) | −0.0029 (14) | 0.0023 (13) |
| C16 | 0.081 (2) | 0.0369 (16) | 0.0588 (17) | −0.0038 (13) | −0.0064 (14) | 0.0094 (13) |
| C17 | 0.077 (2) | 0.0548 (19) | 0.0615 (17) | 0.0054 (14) | 0.0026 (13) | −0.0055 (14) |
| C18 | 0.069 (2) | 0.070 (2) | 0.0638 (17) | 0.0044 (15) | 0.0027 (13) | −0.0025 (15) |
| C19 | 0.090 (2) | 0.0596 (19) | 0.0627 (17) | −0.0049 (16) | 0.0010 (15) | −0.0041 (15) |
| C20 | 0.086 (2) | 0.060 (2) | 0.0706 (19) | −0.0015 (15) | 0.0053 (15) | −0.0064 (15) |
| C21 | 0.106 (3) | 0.067 (2) | 0.076 (2) | 0.0033 (18) | −0.0015 (17) | −0.0039 (17) |
| C22 | 0.102 (3) | 0.074 (2) | 0.077 (2) | 0.0031 (18) | 0.0093 (17) | −0.0084 (18) |
| C23 | 0.152 (3) | 0.077 (3) | 0.080 (2) | 0.015 (2) | 0.004 (2) | −0.0115 (19) |
| C24 | 0.204 (5) | 0.110 (3) | 0.088 (3) | 0.014 (3) | 0.028 (3) | −0.014 (2) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-----------|----------|-----------|
| O1—C6 | 1.376 (3) | C14—C15 | 1.371 (3) |
| O1—C7 | 1.381 (3) | C15—C16 | 1.379 (3) |
| O2—C7 | 1.194 (3) | C15—H15 | 0.9300 |
| O3—C10 | 1.198 (3) | C16—H16 | 0.9300 |
| O4—C10 | 1.347 (3) | C17—C18 | 1.503 (3) |
| O4—C11 | 1.414 (3) | C17—H17A | 0.9700 |
| O5—C14 | 1.378 (3) | C17—H17B | 0.9700 |
| O5—C17 | 1.422 (3) | C18—C19 | 1.505 (4) |
| C1—C6 | 1.363 (3) | C18—H18A | 0.9700 |
| C1—C2 | 1.374 (4) | C18—H18B | 0.9700 |
| C1—H1 | 0.9300 | C19—C20 | 1.510 (3) |
| C2—C3 | 1.381 (4) | C19—H19A | 0.9700 |
| C2—H2 | 0.9300 | C19—H19B | 0.9700 |

| | | | |
|------------|-------------|---------------|-----------|
| C3—C4 | 1.357 (3) | C20—C21 | 1.496 (4) |
| C3—H3 | 0.9300 | C20—H20A | 0.9700 |
| C4—C5 | 1.390 (3) | C20—H20B | 0.9700 |
| C4—H4 | 0.9300 | C21—C22 | 1.502 (4) |
| C5—C6 | 1.386 (3) | C21—H21A | 0.9700 |
| C5—C9 | 1.427 (3) | C21—H21B | 0.9700 |
| C7—C8 | 1.461 (3) | C22—C23 | 1.527 (4) |
| C8—C9 | 1.342 (3) | C22—H22A | 0.9700 |
| C8—C10 | 1.472 (3) | C22—H22B | 0.9700 |
| C9—H9 | 0.9300 | C23—C24 | 1.479 (4) |
| C11—C16 | 1.368 (4) | C23—H23A | 0.9700 |
| C11—C12 | 1.369 (3) | C23—H23B | 0.9700 |
| C12—C13 | 1.375 (3) | C24—H24A | 0.9600 |
| C12—H12 | 0.9300 | C24—H24B | 0.9600 |
| C13—C14 | 1.386 (4) | C24—H24C | 0.9600 |
| C13—H13 | 0.9300 | | |
| C6—O1—C7 | 123.4 (2) | C15—C16—H16 | 120.4 |
| C10—O4—C11 | 121.06 (19) | O5—C17—C18 | 109.0 (2) |
| C14—O5—C17 | 117.82 (19) | O5—C17—H17A | 109.9 |
| C6—C1—C2 | 118.8 (3) | C18—C17—H17A | 109.9 |
| C6—C1—H1 | 120.6 | O5—C17—H17B | 109.9 |
| C2—C1—H1 | 120.6 | C18—C17—H17B | 109.9 |
| C1—C2—C3 | 120.8 (3) | H17A—C17—H17B | 108.3 |
| C1—C2—H2 | 119.6 | C17—C18—C19 | 112.6 (2) |
| C3—C2—H2 | 119.6 | C17—C18—H18A | 109.1 |
| C4—C3—C2 | 119.6 (3) | C19—C18—H18A | 109.1 |
| C4—C3—H3 | 120.2 | C17—C18—H18B | 109.1 |
| C2—C3—H3 | 120.2 | C19—C18—H18B | 109.1 |
| C3—C4—C5 | 120.9 (3) | H18A—C18—H18B | 107.8 |
| C3—C4—H4 | 119.5 | C18—C19—C20 | 115.8 (2) |
| C5—C4—H4 | 119.5 | C18—C19—H19A | 108.3 |
| C6—C5—C4 | 117.9 (2) | C20—C19—H19A | 108.3 |
| C6—C5—C9 | 117.6 (2) | C18—C19—H19B | 108.3 |
| C4—C5—C9 | 124.5 (2) | C20—C19—H19B | 108.3 |
| C1—C6—O1 | 118.0 (2) | H19A—C19—H19B | 107.4 |
| C1—C6—C5 | 121.8 (2) | C21—C20—C19 | 113.8 (2) |
| O1—C6—C5 | 120.2 (2) | C21—C20—H20A | 108.8 |
| O2—C7—O1 | 116.1 (2) | C19—C20—H20A | 108.8 |
| O2—C7—C8 | 127.4 (2) | C21—C20—H20B | 108.8 |
| O1—C7—C8 | 116.5 (2) | C19—C20—H20B | 108.8 |
| C9—C8—C7 | 119.6 (2) | H20A—C20—H20B | 107.7 |
| C9—C8—C10 | 121.6 (2) | C20—C21—C22 | 116.5 (3) |
| C7—C8—C10 | 118.7 (2) | C20—C21—H21A | 108.2 |
| C8—C9—C5 | 122.6 (2) | C22—C21—H21A | 108.2 |
| C8—C9—H9 | 118.7 | C20—C21—H21B | 108.2 |
| C5—C9—H9 | 118.7 | C22—C21—H21B | 108.2 |
| O3—C10—O4 | 123.6 (2) | H21A—C21—H21B | 107.3 |

| | | | |
|---------------|------------|-----------------|------------|
| O3—C10—C8 | 126.3 (2) | C21—C22—C23 | 112.5 (3) |
| O4—C10—C8 | 110.0 (2) | C21—C22—H22A | 109.1 |
| C16—C11—C12 | 121.3 (2) | C23—C22—H22A | 109.1 |
| C16—C11—O4 | 115.6 (2) | C21—C22—H22B | 109.1 |
| C12—C11—O4 | 122.8 (2) | C23—C22—H22B | 109.1 |
| C11—C12—C13 | 119.3 (2) | H22A—C22—H22B | 107.8 |
| C11—C12—H12 | 120.4 | C24—C23—C22 | 115.6 (3) |
| C13—C12—H12 | 120.4 | C24—C23—H23A | 108.4 |
| C12—C13—C14 | 120.3 (2) | C22—C23—H23A | 108.4 |
| C12—C13—H13 | 119.9 | C24—C23—H23B | 108.4 |
| C14—C13—H13 | 119.9 | C22—C23—H23B | 108.4 |
| C15—C14—O5 | 125.3 (2) | H23A—C23—H23B | 107.4 |
| C15—C14—C13 | 119.4 (2) | C23—C24—H24A | 109.5 |
| O5—C14—C13 | 115.2 (2) | C23—C24—H24B | 109.5 |
| C14—C15—C16 | 120.5 (3) | H24A—C24—H24B | 109.5 |
| C14—C15—H15 | 119.7 | C23—C24—H24C | 109.5 |
| C16—C15—H15 | 119.7 | H24A—C24—H24C | 109.5 |
| C11—C16—C15 | 119.2 (2) | H24B—C24—H24C | 109.5 |
| C11—C16—H16 | 120.4 | | |
| | | | |
| C6—C1—C2—C3 | 0.6 (5) | C9—C8—C10—O3 | 148.8 (3) |
| C1—C2—C3—C4 | −1.0 (5) | C7—C8—C10—O3 | −28.5 (4) |
| C2—C3—C4—C5 | 0.7 (4) | C9—C8—C10—O4 | −28.8 (3) |
| C3—C4—C5—C6 | 0.1 (4) | C7—C8—C10—O4 | 153.9 (2) |
| C3—C4—C5—C9 | −178.9 (2) | C10—O4—C11—C16 | −131.1 (3) |
| C2—C1—C6—O1 | −179.0 (3) | C10—O4—C11—C12 | 55.5 (3) |
| C2—C1—C6—C5 | 0.2 (4) | C16—C11—C12—C13 | −0.1 (4) |
| C7—O1—C6—C1 | −179.5 (2) | O4—C11—C12—C13 | 172.9 (2) |
| C7—O1—C6—C5 | 1.2 (4) | C11—C12—C13—C14 | 0.7 (4) |
| C4—C5—C6—C1 | −0.6 (4) | C17—O5—C14—C15 | −11.9 (4) |
| C9—C5—C6—C1 | 178.5 (2) | C17—O5—C14—C13 | 167.5 (2) |
| C4—C5—C6—O1 | 178.7 (2) | C12—C13—C14—C15 | −0.7 (4) |
| C9—C5—C6—O1 | −2.3 (4) | C12—C13—C14—O5 | 179.8 (2) |
| C6—O1—C7—O2 | −176.5 (2) | O5—C14—C15—C16 | 179.5 (2) |
| C6—O1—C7—C8 | 1.8 (4) | C13—C14—C15—C16 | 0.1 (4) |
| O2—C7—C8—C9 | 174.3 (3) | C12—C11—C16—C15 | −0.5 (4) |
| O1—C7—C8—C9 | −3.8 (4) | O4—C11—C16—C15 | −174.0 (2) |
| O2—C7—C8—C10 | −8.4 (4) | C14—C15—C16—C11 | 0.5 (4) |
| O1—C7—C8—C10 | 173.6 (2) | C14—O5—C17—C18 | 178.6 (2) |
| C7—C8—C9—C5 | 2.8 (4) | O5—C17—C18—C19 | 174.9 (2) |
| C10—C8—C9—C5 | −174.5 (2) | C17—C18—C19—C20 | 175.7 (2) |
| C6—C5—C9—C8 | 0.3 (4) | C18—C19—C20—C21 | −178.6 (3) |
| C4—C5—C9—C8 | 179.2 (3) | C19—C20—C21—C22 | −177.7 (3) |
| C11—O4—C10—O3 | −4.6 (4) | C20—C21—C22—C23 | 180.0 (3) |
| C11—O4—C10—C8 | 173.1 (2) | C21—C22—C23—C24 | −178.1 (3) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C4—H4 \cdots O1 ⁱ | 0.93 | 2.59 | 3.513 (4) | 174 |
| C9—H9 \cdots O2 ⁱ | 0.93 | 2.51 | 3.420 (3) | 167 |
| C16—H16 \cdots O2 ⁱⁱ | 0.93 | 2.71 | 3.551 (3) | 151 |
| C16—H16 \cdots O3 ⁱⁱ | 0.93 | 2.63 | 3.338 (3) | 133 |

Symmetry codes: (i) $x, y+1, z$; (ii) $-x, -y+1, -z$.